Sulfur in Elemental Sulfur Formulations and Sulfur Coated Urea

Scope

This method determines the total sulfur in elemental sulfur fertilizers and sulfur coated urea products.

Summary

The sample is washed initially with water and then with sulfur saturated acetone. The sulfur is then extracted from the sample with carbon disulfide. The loss of weight from this treatment is equal to the mass of sulfur present in the sample.

Comments

The three carbon disulfide washes (step G) may not be sufficient to extract all of the sulfur from some high sulfur samples. If a fertilizer is deficient in sulfur, rerun the sample but use additional carbon disulfide washes or larger volume washes and compare the results.

Apparatus and Materials

A. 125 ml erlenmeyer flask, glass stoppered.

B. Gooch crucible.

C. Oven, 100°C.

D. Glass fiber filter paper.

Reagents

A. Acetone, sulfur saturated.

B. Carbon disulfide.

Procedure

A. Accurately weigh a sample containing 200-300 mg of sulfur into a 125 ml glass stoppered erlenmeyer flask. Record the sample weight to the nearest 0.0001 g.

B. Add 50 ml of deionized water, stopper, and shake vigorously for 30 seconds.

C. Filter with suction through a gooch crucible containing glass fiber filter paper. Wash the residue with deionized water.

D. Wash the residue in the gooch crucible with five 10 ml portions of sulfur saturated acetone.

E. Dry the gooch crucible and contents for 1 hour at 100°C.

F. Cool the gooch crucible and contents in a desiccator and weigh to the nearest 0.0001 g.

G. Wash the residue in the gooch crucible with three 5 ml portions of carbon disulfide and drain. Carbon disulfide is flammable and toxic so do the washings in a well ventilated hood.

H. Dry the crucible and contents for 1 hour at 100°C.

I. Cool the crucible and contents in a desiccator and weigh to the nearest 0.0001 g.

Calculations

% Sulfur = (weight of crucible and contents before washing with carbon disulfide) - (weight of crucible and contents after washing with carbon disulfide) x 100 ÷ sample weight in grams

Quality Control

A. Check the temperature of the drying ovens before use with a calibrated thermometer and document on worklist.

B. Run blanks when specified in the method.

C. Monitor time.

1. Acid extraction of sulfate sulfur in dry fertilizer should be done for about 10 minutes.

2. Digestion of B_2SO_4 precipitate on steam bath or low temperature hot plate should be done for about one hour.

3. Drying of B_2SO_4 precipitate should be done for one hour at 250^{o}C.

4. Residue containing elemental sulfur should be dried one hour at 100^{o}C before and after extraction of the sulfur.

5. Liquid samples should be refluxed for one hour during the oxidation with H_2O_2.

Bibliography

Official Methods of Analysis (1984) 14th Ed., AOAC, Washington, D.C., sec. 2.183(c)